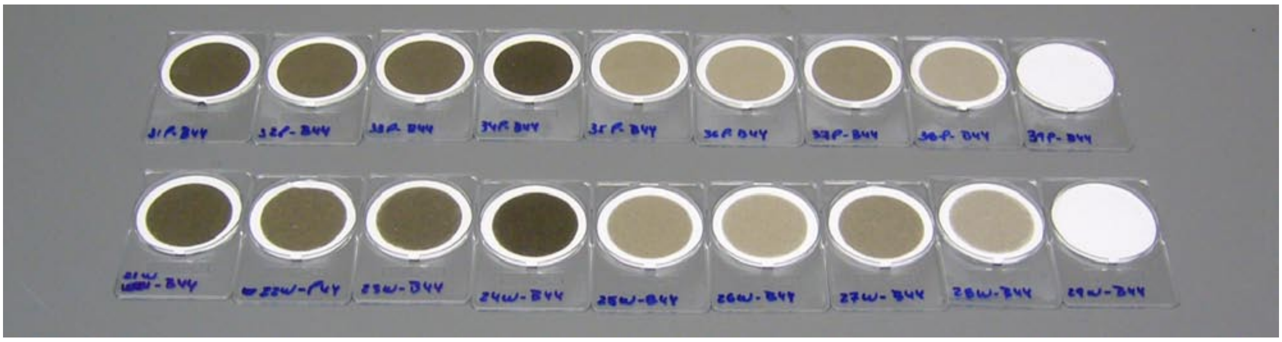
European Commission

JRC TECHNICAL REPORTS



Determination of Particulate Matter according to CEN and EMEP standards at the Atmosphere Biosphere and Climate-Integrated Station (ABC-IS), Ispra (IT)

Claudio A. Belis
Valerio Pedroni
Jose Cancelinha
Annette Borowiak

2012

Report EUR 25802 EN

European Commission
Joint Research Centre
Institute for Environment and Sustainability

Contact information
Claudio A. Belis
Address: Joint Research Centre, Via Enrico Fermi 2749, TP 441, 21027 Ispra (VA), Italy
E-mail: claudio.belis@jrc.ec.europa.eu
Tel.: +39 0332 786644
Fax: +39 0332 785236

http://ies.jrc.ec.europa.eu/
http://www.jrc.ec.europa.eu/

This publication is a Technical Report by the Joint Research Centre of the European Commission.

JRC79144

EUR 25802 EN

ISBN 978-92-79-28424-3 (pdf)

ISSN 1831-9424 (online)

doi: 10.2788/8320

Luxembourg: Publications Office of the European Union, 2013

Printed in Italy

Introduction

From May 2011 to December 2012, samples of PM_{10} were collected in the Atmosphere, Biosphere, Climate – Integrated Station (ABC-IS) using different instruments in parallel. These samples were collected to perform the chemical characterization of PM_{10}, especially those species that are not analyzed routinely. These samples were used for a study on the levels of trace elements (Cavalli et al, 2012) and the comparability of two analytical methods to determine trace elements in PM_{10} (Yatkin et al., 2012). In addition, a thorough chemical characterization of the PM_{10} collected in this monitoring campaign is planned to be used as input for a receptor model exercise to identify the sources of PM in this site. The objective of the present report is to describe the tests aiming at ensuring the quality of the measurements and the comparability between the different sampling methodologies .

Sampling equipment

In the ABC-IS PM_{10} samples are collected using Partisol samplers in conformity with the US-EPA reference method and are weighed after conditioning at 20% relative humidity (Table 1). Other samples were collected following the same scheme with samplers FAI Hydra and Leckel SEQ 47/50, which are compliant with the EN 12341 standard of the European Committee for Standardization (CEN) (Table 1). The SEQ 47/50 sampling system has been operated in Ispra building 44, approximately 450 m away from the ABC-IS sampling site.

The FAI Hydra is a dual sampling line instrument. For this monitoring campaign these sampling lines were fitted with two different types of filters: Quartz Pallflex tissuequartz 2500QAT and Teflon Pall Teflo ®, whereas the SEQ system was operated with quartz filters only.

In order to balance the sampling and analytical effort with the time resolution and data coverage , 24 hour PM_{10} samples were collected from 8.00 UTM to 8.00 UTM every 6th day, matching the sampling scheme in place for the standard EMEP PM_{10} parameters. By selecting this setup it was expected to approximate annual average values and seasonal trends of PM_{10} concentration and chemical composition.

Table 1. Technical characteristics of the equipment used for PM_{10} sample collection

	FAI Hydra Sequential Gravimetric Dual Sampler. PM10 inlet according to CEN EN12341 Dual channel sequential sampling Sampling flow-rate 38.3 l/min Two filters contemporaneously (1 quartz filter and 1 teflon filter) every sixth day
	2 LECKEL SEQ 47/50 equipped with PM10 inlet according to CEN EN 12341, one of which with scrubber Sampling flow-rate 38.3 l/min 1 quartz filter every sixth day
	PARTISOL PLUS 2025 U.S. EPA PM-2.5 and PM-10 reference method designation Sequential sampling with a 16-filter capacity Sampling flow-rate 16.7 l/min 1 quartz filter every sixth day

Weighing facilities and methods

The gravimetric determinations were carried out in two different facilities. One of them, located in Ispra building 44, operates in conformity with the cited EN standard 12341 while the other facility, located in building 29, is operated according to a protocol that has been developed within the framework of the EMEP programme. The used methods are described more in detail in the section below.

<u>CEN WEIGHING METHOD</u>

Filters are pre-conditioned for 48 hours in an air-controlled room with a temperature of 20 ± 1 ºC and a relative humidity of 50 ± 3 %. Weighing is performed with an analytical balance (METTLER TOLEDO AX26) connected to a PC that stores automatically the measurements (Figure 1). This balance is calibrated according to the Italian national calibration system (SIT) standards in order to assure the traceability of the measurements to a primary standard and estimate the weighing uncertainty.

Figure 1. CEN weighing facility

<u>EMEP WEIGHING METHOD</u>

Filters are pre-conditioned for 24 hours in a glove controlled box at a temperature of 20 ± 2 ºC and a relative humidity of 20 ± 5 %. The relative humidity is controlled by an automated system that controls a dry air input valve (Figure 2). Weighing is performed with an analytical balance (Satorius MC5).

Figure 2. EMEP weighing facility

Quality checks

In order to assure the quality of the measurements the following quality controls were performed:

Flow rate check: monthly.
Field blanks in every sampler: monthly.
Comparison between balances Mettler AX26 and Sartorius MC5 using same weights: annually.
Comparison between CEN vs EMEP gravimetric methods, intensive campaign: 03/12/2011 ÷ 15/12/2011.
Comparison between CEN-sampling EMEP weighing vs. CEN, intensive campaign: 10/10/2012 ÷ 19/10/2012 .

Balance intercomparison

A comparison of the performance of the two balances used in each weighing facility was carried out. For that purpose the mass of the same reference weights was determined 10 times in each facility. The observed masses obtained with the two balances were comparable and standard deviations of both weighing systems were well below 1 µg (Table 2).

Table 2 Balance intercomparison

10 replicates	Mettler AX26	Sartorius MC5
weight mg	100	100
standard deviation	0,000843	0,000675
weight mg	50	50
standard deviation	0,000632	0,000789

Flow rate checks

As specified in the EN standard 12341, deviations in the actual flow rate from the nominal flow rate were kept under control. The instruments in conformity with CEN have warning systems for these parameters and register day and time of irregular values. In order to check possible drifts in the instrument flow-meters a flow rate check is performed every month on the instrumentation using a certified flow-meter. The results of these tests, reported in table 3, confirm that sampling flow was stable and within the EN12341 requirements.

Table 3. Flow rate check

	Fai Q	Fai T	Leckel 66	Leckel 68
units	l/min	l/min	l/min	l/min
April	38,2	38,3	38,2	38,3
May	38,2	38,2	38,2	38,2
June	38,2	38,2	38,2	38,2
July	38,2	38,2	38,3	38,2
August-September	38,1	38,1	38,2	38,3
October	38,2	38,3	38,2	38,4
November	38,4	38,2	38,2	38,2

Field blanks

Field blanks are filters that undergo the same process as sampled filters with the only exception that they are not exposed to ambient air flow. These filters are useful to assess the influence of filter storage and transportation before, during and after sampling. However, only partial information on sampling artifacts can be obtained with this method. This test was introduced from the beginning of the monitoring campaign for FAI samplers and subsequently was applied to the Leckel instruments. Field blank masses in the Leckel samplers were on average higher than those observed in FAI samplers (table 4). In the FAI sampler field blanks were almost always below the threshold of 60 µg (including both quartz and Teflon filters) while those in the Leckel samplers were almost always above that value. The cause of this difference is under investigation.

Table 4. Field blank mass

	Leckel 66	Leckel 68	FAI Q	FAI T
units	µg	µg	µg	µg
July 2011			43,5	0,5
August 2011			16	10
September 2011			49	56
October 2011			23,5	
November 2011			23	58,5
December 2011			10,5	42,3
January 2012				49,5
February 2012		85,5	12	16
March 2012		5		33
April 2012	85,5	57	15	10,5
May 2012	128	67,5	35	43
June 2012	103,5	92	66,5	48
July 2012	95,5	131,5	98,5	
August 2012	110	98	43	14,5
September 2012	142,5	140,5	65	10

Time trend of PM_{10}

PM_{10} sampling started on 25/5/2012 with one 24 hour sample collected from 8.00 UTM to 8.00 UTM every 6^{th} day to match the sampling scheme in place for the standard EMEP PM10 parameters. In order to obtain specific information concerning comparison of different systems or periods of particular interest three intensive campaigns with everyday sampling were carried out in the following time windows:

Intensive campaign 1: 03/12/2011 ÷ 15/12/2011
Intensive campaign 2 : 10/10/2012 ÷ 19/10/2012
Intensive campaign 3 : 30/11/2012 ÷ 21/12/2012

In figure 3 is reported the average trend of PM_{10} recorded from May 2011 to November 2012. The lowest levels are those observed during the warm season (from April to August) while the highest levels are those observed in the cold season, especially in early and late winter. This temporal pattern fits the main pollution episodes observed in $PM_{2.5}$ which is sampled daily in this site.

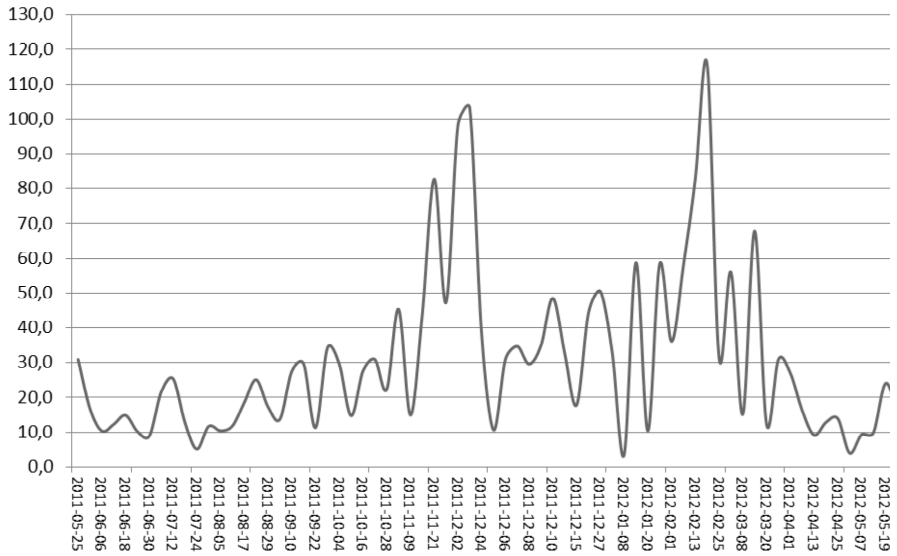


Figure 3. Average trend of PM_{10} from 25/5/2011 to 9/11/2012 (µg/m³)

Comparisons between sampling and weighing protocols

The PM_{10} concentrations measured with the two lines of the FAI Hydra using different types of filters are quite comparable (r^2= 0.99). Differences are within 2% and the intercept is close to 1 µg/m^3 (Figure 3). Similar results are observed when comparing the two Leckel samplers (with and without scrubber).

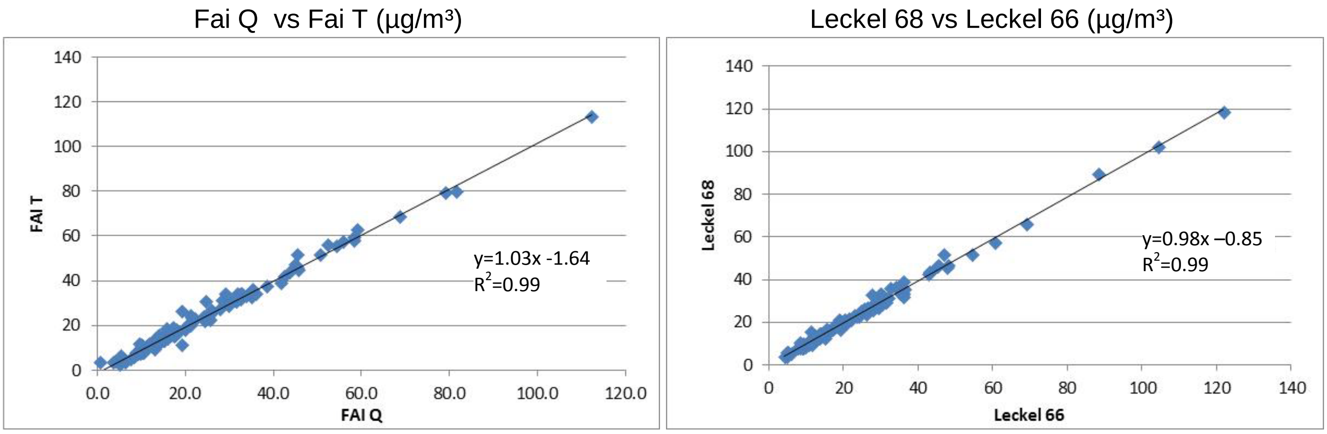


Figure 3. Comparison of replicate measurements in the same type of instrument using orthogonal regression and the coefficient of determination.

Differences are more evident when comparing concentrations obtained with different sampling and weighing methods. The PM_{10} concentrations obtained with the FAI Hydra (CEN protocol) are 4% higher than those obtained with the Partisol (EMEP protocol). However at concentrations below 60 µg/m^3, the values of FAI Hydra tend to be lower than those of the Partisol (figure 4). Same pattern is observed between Leckel and Partisol with differences in this case arriving at 10% and the intercept being quite negative.

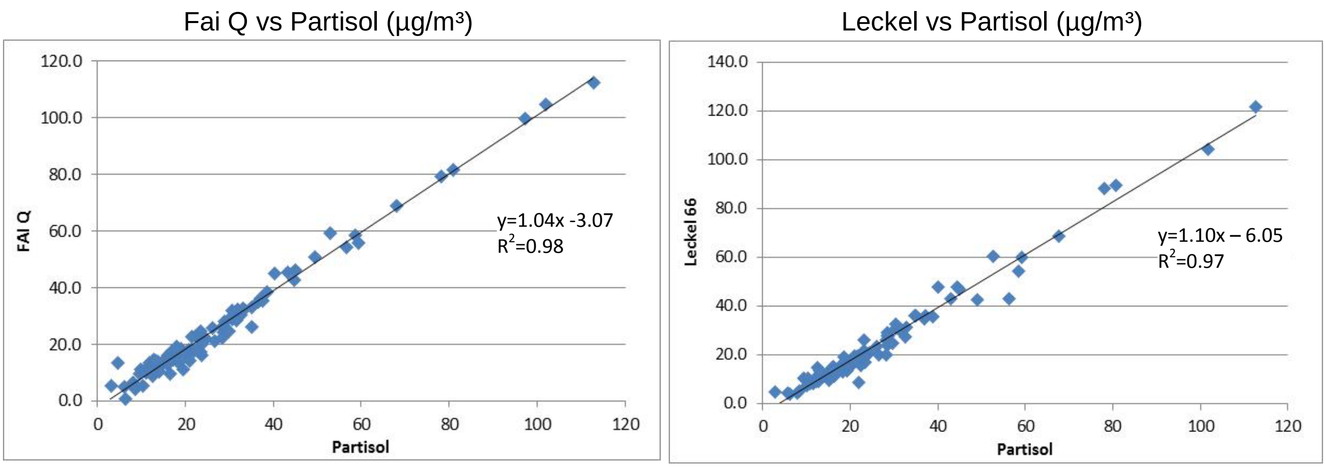


Figure 4. Comparison of measurements obtained with different gravimetric protocols instrument using orthogonal regression and the coefficient of determination.

Comparing instruments with different operating principles is even more critical. The scatter plots representing the comparison between gravimetric determinations (FAI) and concentrations obtained with TEOM FDMS, an automated measurement system, are shown in figure 5. The dispersion of the data around the regression line is higher and the coefficient of determination is ca. 0.89.

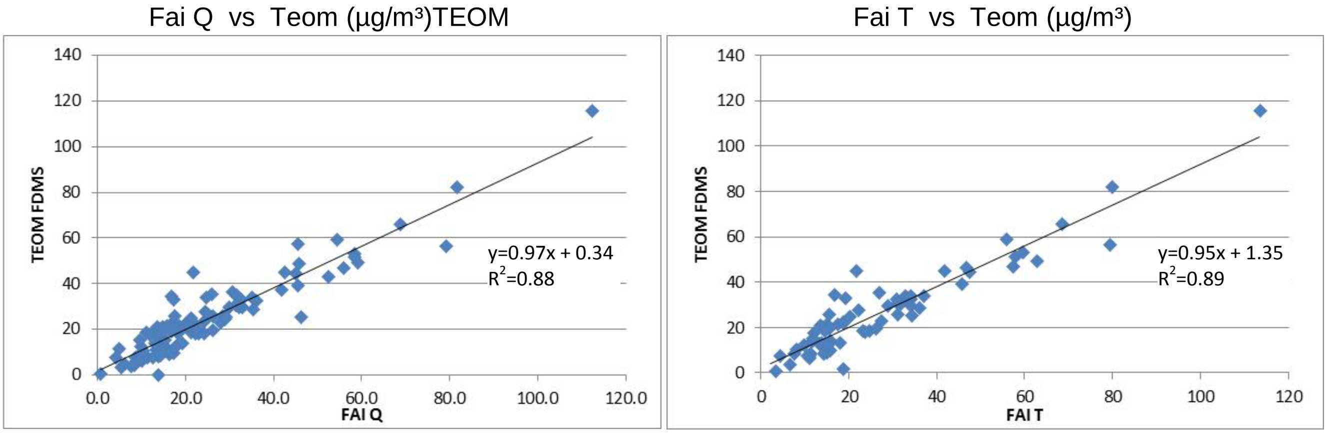


Figure 5. Comparison of PM10 concentrations obtained with different operating principles instrument using orthogonal regression and the coefficient of determination.

The values of the automated method are ca. 3-5 % lower than those of the gravimetric method with an intercept of ca. 2 µg/m^3. However, it must be taken into account that the detection limit for TEOM FDMS is 5 µg/m^3.

Similar regression parameters are observed for gravimetric measurements obtained with other instrument and other gravimetric protocol (Partisol). Almost 10% systematic underestimation of the Teom compared with the gravimetric Leckel is observed, which is compensated at low concentrations by a relatively high slope (ca. 3 µg/m^3) (Figure 6).

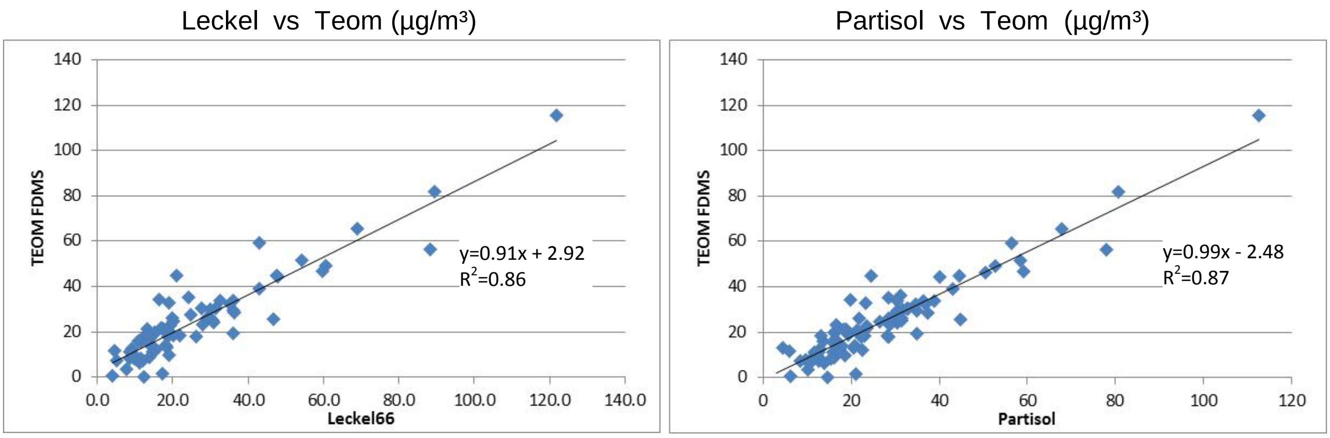


Figure 6. Comparison of PM_{10} concentrations obtained with different operating principles instrument using orthogonal regression and the coefficient of determination.

Concluding remarks

The monitoring campaign 2011-2012 at the ABC-IS monitoring network provided sound information about the levels of PM_{10} in the area. The results of the balance and flow rate quality check were satisfactory. The uncertainty between replicate samples collected with the same instrument fell within 2%. Higher differences were observed between samples collected with different gravimetric methods, especially at concentrations above 60 µg/m^3. The greatest differences were those observed between measurements obtained with the gravimetric methods and the automated method based on oscillating microbalance (TEOM FDMS). This automated method presented values lower than those obtained with the gravimetric methods, with the exception of low concentrations where the uncertainty of the automated method is higher.

The positive results of the quality tests and the good comparability between the different gravimetric methods, especially for concentrations below 60 µg/m^3 suggest that the samples are fit for subsequent chemical characterization and their use for receptor modelling purposes.

References

Cavalli F. R. Passarella and V. Pedroni. 2012. Observations of trace elements in particulate matter in Ispra: 2011-2012 data. JRC technical report.

EN 12341, 1998. Determination of the PM10 fraction of suspended particulate matter - Reference method and field test procedure to demonstrate reference equivalence of measurement methods.

S. Yatkin, C. A. Belis, J. Cancelhina, F. Cavalli, R. Passarella, V. Pedroni, and M. Gerboles.2012. Assessment of comparability of heavy metal determined by EDXRF and ICPMS at the JRC EMEP station. JRC technical report.

European Commission
EUR 25802 – Joint Research Centre – Institute for Environment and Sustainability

Title: Determination of PM according to CEN and EMEP standards at the Atmosphere Biosphere and Climate-Integrated Station (ABC-IS), Ispra (IT)

Author(s): Claudio A. Belis, Valerio Pedroni, Jose Cancelinha, Annette Borowiak

Luxembourg: Publications Office of the European Union

2013 – 12 pp. – 21.0 x 29.7 cm

EUR – Scientific and Technical Research series – ISSN 1831-9424 (online)

ISBN 978-92-79-28424-3 (pdf)

doi: 10.2788/8320

Abstract

From May 2011 to December 2012, samples of PM_{10} were collected in the Atmosphere, Biosphere, Climate – Integrated Station (ABC-IS) using different instruments in parallel. The samples were collected to be used for chemical characterization of the PM_{10}. The objective of the present report is to describe the tests aiming at ensuring the quality of the measurements and the comparability between the different sampling methodologies. The positive results of the quality tests and the good comparability between the different gravimetric methods, especially for concentrations below 60 µg/m^3 suggest that the samples are fit for subsequent chemical characterization and their use for modelling purposes.

LB-NA-25802-EN-N

As the Commission's in-house science service, the Joint Research Centre's mission is to provide EU policies with independent, evidence-based scientific and technical support throughout the whole policy cycle.

Working in close cooperation with policy Directorates-General, the JRC addresses key societal challenges while stimulating innovation through developing new standards, methods and tools, and sharing and transferring its know-how to the Member States and international community.

Key policy areas include: environment and climate change; energy and transport; agriculture and food security; health and consumer protection; information society and digital agenda; safety and security including nuclear; all supported through a cross-cutting and multi-disciplinary approach.

Publications Office

ISBN 978-92-79-28424-3